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Fiber Bragg grating applied pulsed photoacoustic detection technique for online monitoring concentration of liquid

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The fiber Bragg grating (FBG) applied pulsed Photoacoustic (PA) detection technique for the online monitoring concentration of liquid is proposed. Due to its adoption, a sensor based on this technique not only measures consistency but also temperature and flow speed at the real-and-same time, i.e., enabling the realization of a multi-functional optical fiber sensor. The advantage of the PA technique is that absorbed energy is converted exclusively into heat without generating PA signals caused by scattering particles. The measurement system and its design method are described. Based on the results of calculation for designing sensing system, we optimize the relations among linearity, sensitivity, stability, and portability. Sensing linearity is shown within a concentration range from 5 to 150 ppm with a Rhodamine 6G solution.

1 Introduction

The real-time monitoring of the concentration of raw liquids and wastewater flow in ducts is one required process control in chemical plants. The development of such an online measurement technique has been desired. Previously, a measurement technique based on optical transmission was widely adopted as the classical technique because sufficient measurement of the dynamic range and accuracy can be obtained, even though the technique is quite primitive. Nevertheless, light-scattering particles included in the fluid, such as bubbles, scales, and colloidal solutions strongly affect the transmitted light intensity, not only dramatically reducing the dynamic range but also greatly increasing the measurement errors.

To overcome this problem, a photoacoustic (PA) sensor system was previously proposed for the online monitoring of highly concentrated and opaque liquid samples [1]. In this system, a pulsed PA spectroscopy is based on the absorption of short laser pulses on the inside of the condensed matter [1]-[3]. The advantage of the PA technique is that absorbed energy is converted exclusively into heat without generating PA signals caused by scattering particles; in other words, the affects of the parasitical phenomena caused by such radiative relaxation processes as fluorescence and phosphorescence can be neglected [1],[2]. In addition, PA spectroscopy allows us to determine the concentration over several orders of magnitude without dilution even if the measurement object is opaque. Even though this sensor system is unique, the following fatal weakness exists. This PA sensor is not applicable to acidic fluids due to the maintenance required to sustain the performance caused by the metal erosion of electrodes from acidity. Besides, we cannot perfectly shield this sensor due to the transducer structure to avoid electro-magnetical noise, even though the PA (electrical) signal, which is transduced with a piezo device, is a slight signal under a few μA . Because of these, the actual use of this sensor in chemical plants is unsuitable.

To overcome this problem, we propose a fiber Bragg grating (FBG) [4]-[7] applied pulsed PA detection technique for the

online monitoring of liquid concentration. In our technique, an optical fiber made of quartz is used for the PA signal generation and detection, and a 0.6-mm-diameter step index (SI) fiber and FBG composed of a single mode (SM) fiber are used as objects, respectively, so our metal-free sensor head is not only small but also maintenance-free. Note that our proposed sensor is substantially noise proof without electro-magnetical shielding, enabling actual use in plants. Furthermore, an FBG applied pulsed PA detector was previously reported, but it was not endowed with sufficient performance, as is our sensor [8].

First, we describe the measurement system as well as how to design the sensor head. Then, the concentration of dye dissolved in the distilled water was measured to estimate capability. Finally, we propose a construction method of a multi-functional sensor applicable to the simultaneous measurement of the concentration, temperature, and speed of the flowing fluid.

2 Measurement system

Figure 1 shows a block diagram of the measurement system. A Q-switched frequency doubled Nd:YAG laser ($\lambda=532\text{ nm}$, Continuum, Minilight) with a repetition rate of 10 Hz was used for the PA excitation beam source. The pulsed beam was focused using a plano-convex lens ($f=150\text{ mm}$), guided into the 0.6-mm-diameter SI fiber, and finally fed to the sensor head. The laser energy can be adjusted with an optical attenuator installed in the laser. The broad beam from the amplified spontaneous emission (ASE) light source with a wavelength of 1530 to 1570 nm was fed to the FBG sensor through the optical circulator, and the beam, reflected with the FBG, was returned to the circulator and fed to an InGaAs pin photo diode (PD) for detecting the optical amplitude through the optical band-pass filter (BPF). Here, the full-width at half-maximum (FWHM) of both the FBG and the BPF as well as the difference between the wavelength center of FBG λ_{FBG} and BPF λ_{BPF} are significant parameters for determining the sensing characteristics. Furthermore, we describe how to determine in optimum these parameters in the next section.

3 Design of FBG sensor

Pulsed PA spectroscopy is based on the absorption of short laser pulses inside the condensed matter. Due to the thermal expansion of the medium, generated pressure waves can be detected by FBG fibers [8]. Here, the energy absorbed inside an irradiated medium with optical absorption coefficient α of the specimen is given by [1],[2]:

$$Q_{(l)} = Q_0 [1 - \exp(-\alpha l)], \quad (1)$$

where Q_0 is the laser energy and l is the distance from the specimen surface along the laser beam (i.e., the above “gap” d). Next, in the case of $\alpha l \ll 1$, Eq. (1) can be approximated by a primary liner function, and amplitude p of the PA signal can be introduced by:

$$p \propto (\beta c^2 / C_p) Q_0 \alpha, \quad (2)$$

where β is the thermal expansion coefficient, c is the speed of sound, and C_p is the heat capacity.

Depending on the optical absorbance of the medium, different PA waveforms are obtained [9]. For weakly absorbing samples, a cylindrical wave, which propagates perpendicularly to the laser beam, is formed because the volume irradiated by a collimated beam is approximately cylindrical. On the other hand, when optical absorbance increases (i.e., in cases of highly absorbing and opaque samples), spherical waves are generated because the optical penetration depth and irradiated volume decrease. Accordingly, even if the absorption coefficient varies over several orders of magnitude, based on Eq.(2), linearity between the PA signal amplitude and the sample concentration can be obtained even though different waveforms are generated. From these relations, the linearity of detection must be obtained with a wide range of acoustic intensity as the condition for designing the sensing element by the following procedure.

Figure 3 shows the numerical calculation results of the relationship between the difference in the center wavelength of FBG with BPF $\Delta\lambda$ (i.e., $\Delta\lambda = |\lambda_{\text{FBG}} - \lambda_{\text{BPF}}|$) and the estimated intensity of the detection signal as the function of the FWHM of FBG. In this calculation, a Gaussian profile, associated with FWHM, was estimated as the band-pass characteristic curve for FBG and BPF. Here, λ_{FBG} is proportional to the length of the mechanical elasticity of FBG, which is independently proportional to each strain by the temperature changing and the acoustic wave. Accordingly, λ_{FBG} involves the information of both the temperature and the strain. In other words, we can measure the consistency of the liquid by detecting the alternate $\Delta\lambda$ value corresponding to the PA signal intensity and the temperature of the liquid in the specimen

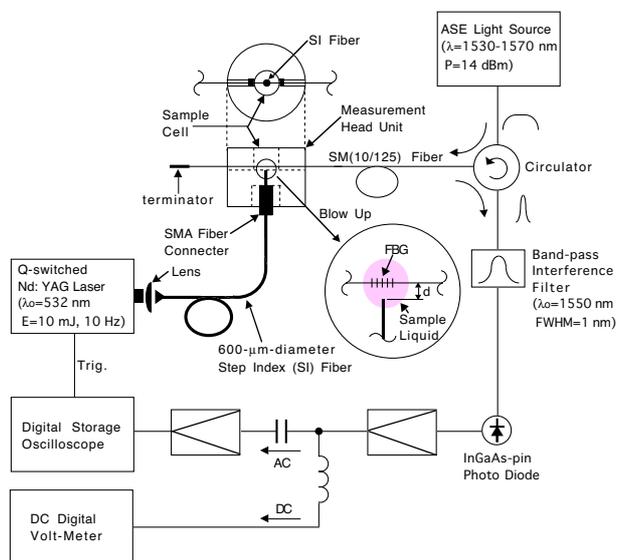


Fig.1 Block diagram of measurement system.

The sample liquid is inserted in the gap d (shown in Fig. 1) between the FBG fiber (i.e., the sensing element of the PA signal) and the SI fiber for exciting the pulsed PA signal assembled with the measurement head unit made of aluminum, whose volume of filled liquid is under 0.1 ml. This gas, which is one important parameter for our sensor technique, will be discussed later. Furthermore, the specimen cell must be made of quartz to avoid metal erosion from the acidity.

The signal detected by the PD was recorded by a digital storage oscilloscope (Agilent Infiniium 54831B) with a time-resolved mode after the signal was amplified. The PA signal was averaged 100 times with an oscilloscope due to the enhancement of the signal-to-noise ratio. Figure 2 shows an example of a recorded typical wave form. The spire waveform with positive and negative polarity, which corresponds to the PA signal, was obviously confirmed. An indefinite waveform was also observed, induced by the nonessential acoustic waves reflected in the multiplex on the inside of the sample vessel. We believe that this facility can only pick up the PA signal due to the time-resolved signal.

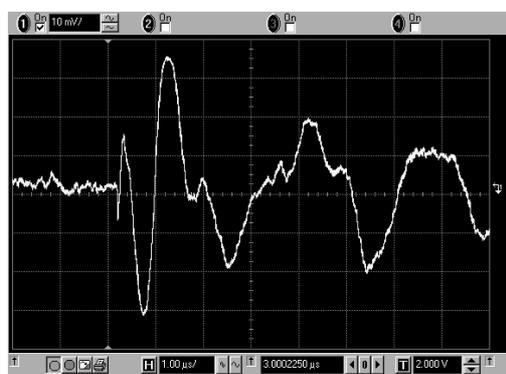


Fig. 2 Typical wave form observed with oscilloscope. Sweep time: 1μs/div, laser wavelength & energy: 532 nm & 1 mJ, consistency of Rhodamine 6G: 50 ppm, solvent: distilled water, FWHM of FBG & BPF: 0.5 & 1 nm, offset $\Delta\lambda$: 1 nm, gap d : 0.1 mm.

cell with the offset DC signal, shown in Fig. 1. Due to these matters, the linearity in Fig. 3 is extremely significant for optimum sensor design. Besides, the differential coefficient on the curve shown in Fig. 3 is also an extremely crucial parameter to determine the sensitivity of the FBG sensor. Based on the calculation results shown in Fig. 3, in this work we set the FWHMs of FBG and BPF to 0.5 and 1 nm, respectively, and adjusted $\Delta\lambda$ to 1 nm as the offset, enabling the optimization of relations among linearity, sensitivity, stability, and portability.

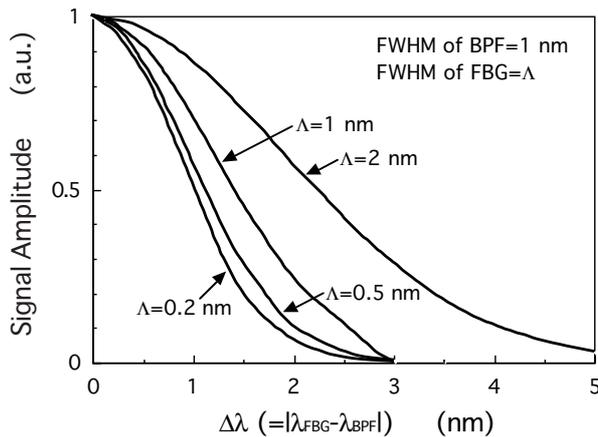


Fig. 3 Numerical calculation results of relationship between $\Delta\lambda$ and estimated intensity of detection signal. FWHM of BPF: 1 nm.

4 Experimental results and discussions

The concentration of dye (Rhodamine 6G, which has powerful optical absorption at 532 nm) dissolved in distilled water was measured to estimate the capability of our technique. Figure 4 shows the concentration dependence of the detected signal amplitude (i.e., PA signal) at $T=287$ K as a function of gap d shown in Fig. 1 (mentioned in Section 2). The laser energy with a fiber-out was controlled to approximately 1 mJ to avoid breakdown of the dye particles. The solid line in this figure means a primary linear function. As seen in this figure, we confirmed that sensing linearity could be maintained within a concentration range from 5 to 150 ppm when gap d was set to 0.1 mm and also estimated that the “limit of detection” (LOD) value is 5 ppm.

On the other hand, sensing linearity could be obtained even through d was set to 0.7 and 1.6 mm. The linearity maintained concentration range, however, narrowed; especially the saturation signal amplitude became low as d widened for the following reason. The effects of the spherical waves became more and more conspicuous as d was widened for the above reason. Here, the attenuation of the spherical waves against the transmission distance has more radical property than the

cylindrical wave. Accordingly, the optical absorption of the dye solution is saturated with low consistency as d is widened.

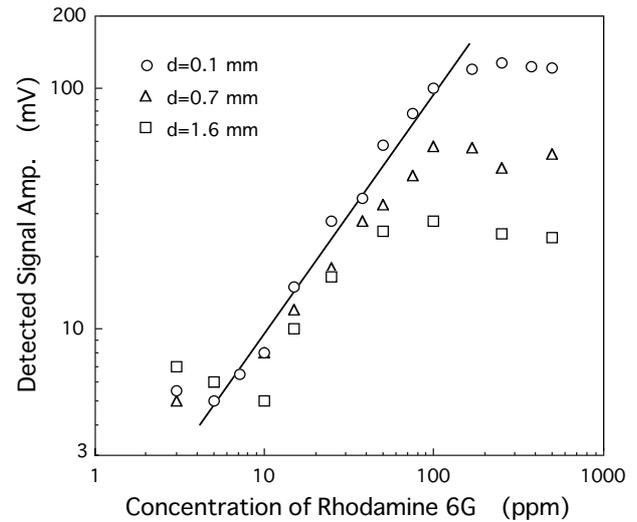


Fig. 4 Concentration dependence of detected signal amplitude. Laser wavelength & energy: 532 nm & 1 mJ, solvent: distilled water, FWHM of FBG & BPF: 0.5 & 1 nm, offset $\Delta\lambda$: 1 nm.

Figure 5 shows the relationship between the specimen temperature and the detected DC voltage shown in Fig. 1 in the case without excitation. The DC voltage is dependent on the temperature. Of course, this result has already been applied in commercial instruments for temperature sensing. Here, since the sonic speed inside the matter greatly depends on the temperature, it is desired to measure the specimen concentration and its temperature synchronously in real-time because of calibration of sonic speed for compensating the PA signal [ref. Eq. (2)]. However, the above recently proposed PA concentration sensor does not essentially function as a thermometer, so instrumental error in concentration measurement increased because the temperature of specimen cannot be compensated for while the specimen liquid is flowing.

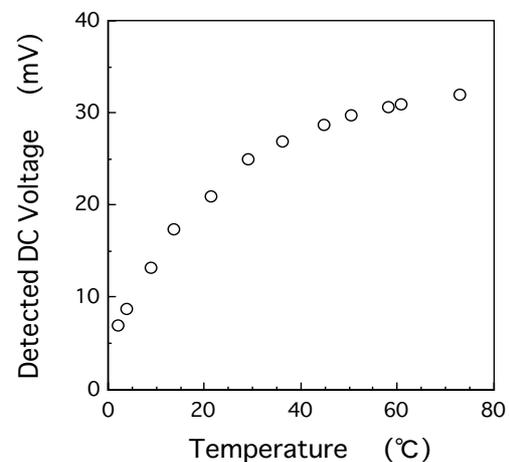


Fig. 5 Specimen temperature dependence of offset DC voltage measured with DC volt-meter shown in Fig. 1. FWHM of FBG & BPF: 0.5 & 1 nm, offset $\Delta\lambda$: 1 nm.

The sensor element (FBG) of our PA technique is very small (under 100 μm in length, ten μm in diameter) compared with the previous PA technique (approximately 10 mm in diameter). Thus, the waveform shown in Fig. 2 becomes broad as the liquid flow speed increases. In other words, we believe that flow speed can be measured by waveform analysis.

5 Summary

We proposed an FBG applied pulsed PA detection technique for the online monitoring concentration of liquid. Our proposed sensor is substantially noise proof without electromagnetic shielding, enabling actual use in plants. We confirmed that sensing linearity can be maintained within a concentration range from 5 to 150 ppm when the concentration of Rhodamine 6G dissolved in the distilled water was measured. We also proposed a technique for synchronously measuring the specimen concentration and temperature in real-time. This is very effective for constructing a multi-functional sensor.

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